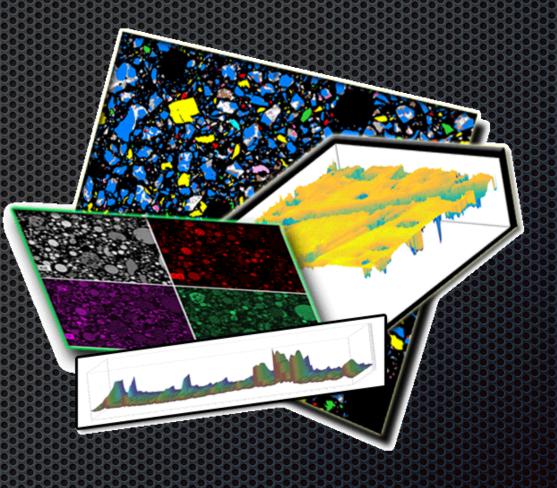
## Using Characterization Data to Model Microstructure & Properties

### Jeff Bullard





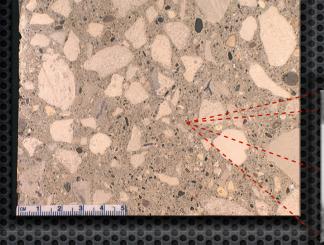




## Background

We want to understand and predict the properties of concrete materials, including its early-age performance and service life







Performance of concrete depends on the properties of the cement binder component

### Properties of cement binder

- change continuously in time
- 0 depend on its proportioning, chemistry, microstructure, and nanostructure

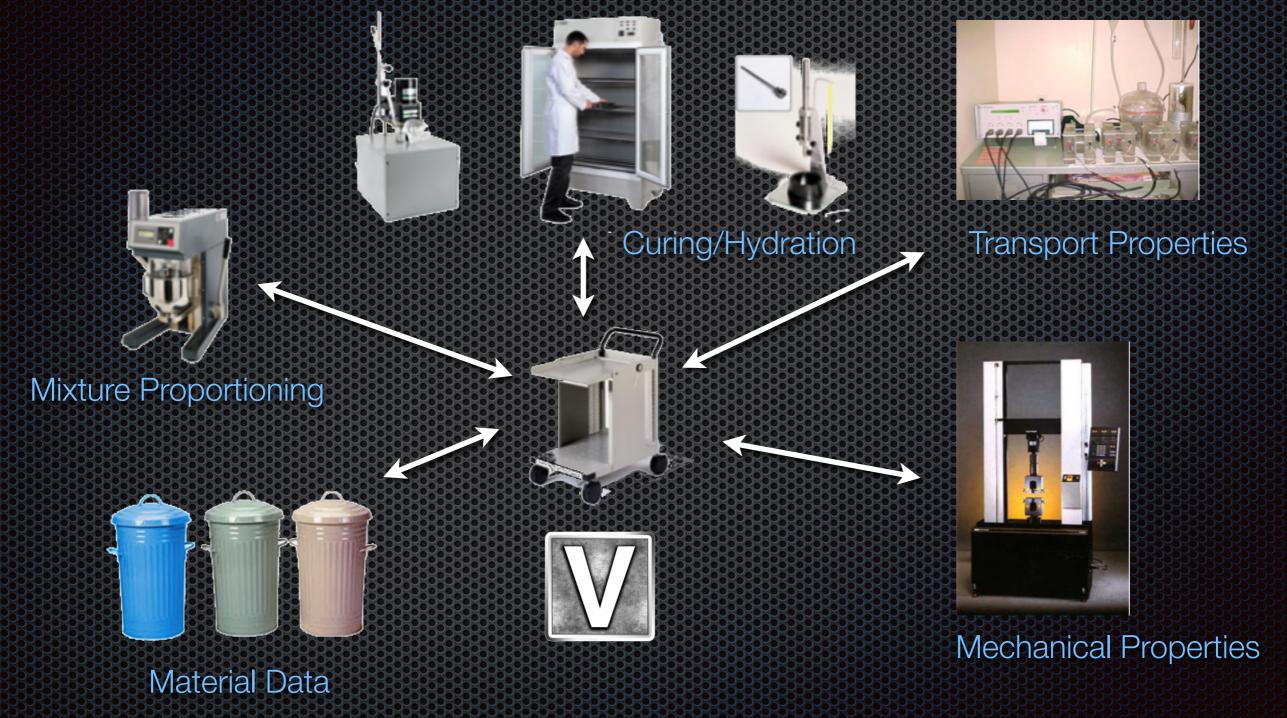
Waste-stream materials







Water



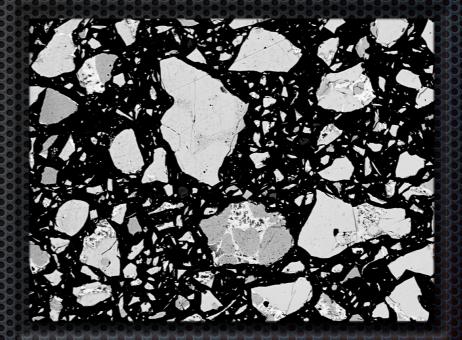
Cement Characterization Workshop, 2018

NS

## **Digital Image Modeling**

What we would like to reproduce accurately in 3D image:

- Phase volume fractions
- Phase surface area fractions
- Phase correlations in space
- Particle size distribution
- <sup>8</sup> Particle shape, if available

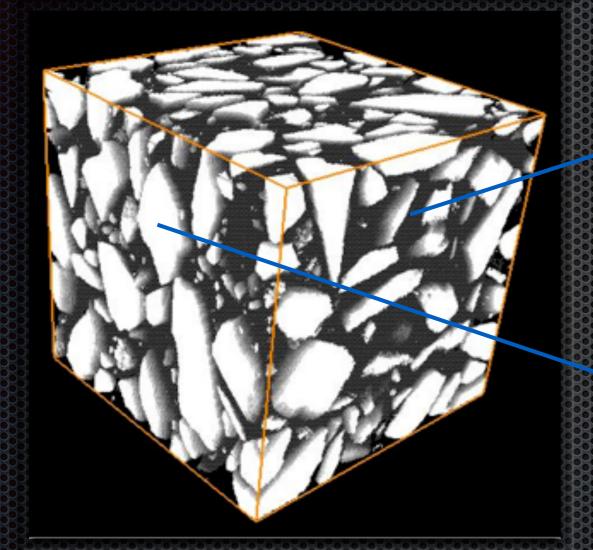


From a 2D image?



## **3D** Particle Shape

X-ray µCT:



E.J. Garboczi and J.W. Bullard, *Cem. Concr. Res.* 34 [10] 1933-1937 (2004)



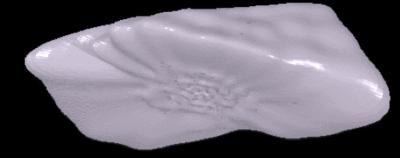
Extract individual particles



## **3D** Particle Shape

Shape characterized by spherical harmonic analysis:





E.J. Garboczi, *Cem. Concr. Res.* **32** [10] 1621-1638 (2002)

(or any engineering math book)

$$r(\theta, \phi) = \sum_{n=0}^{\infty} \sum_{m=-n}^{n} a_{nm} Y_n^m(\theta, \phi)$$
Legendre polynomials
$$Y_n^m(\theta, \phi) = \sqrt{\frac{(2n+1)(n-m)!}{4\pi(n+m)!}} P_n^m(\cos(\theta)) e^{im\phi}$$

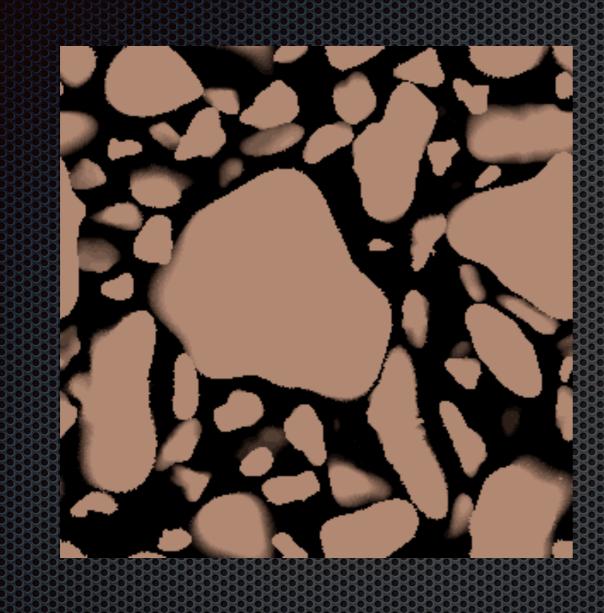
$$a_{nm} = \int_0^{2\pi} \int_0^{\pi} d\phi \, d\theta \, \sin(\theta) \, r(\theta, \phi) \, Y_n^{m*}$$

Solve for SH coefficients, and store in a database for thousands of particles

Retrieve the set  $\{a_{nm}\}$  for a given particle shape and choose arbitrary Euler angles  $\theta, \phi$  to reproduce the particle using the top equation

# NS

## **3D Particle Shape**



Reproduces desired shape distribution

Reproduces desired size distribution

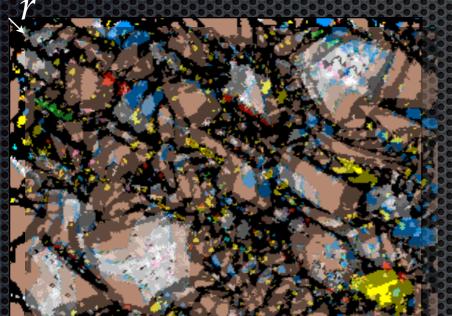
Reproduces desired solid volume fraction, usually to within  $\pm 10^{-4}$ 

Next: Multiphase particles



We want to distribute the clinker phases among particles in a spatially realistic way, at least statistically.

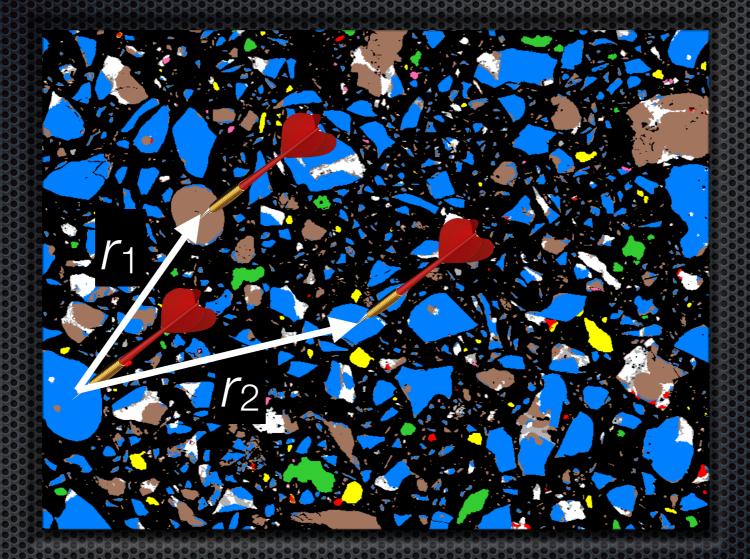
To do so, first measure two-point correlation functions on different phases or collections of phases in segmented SEM image:



$$f_{2,i}(r) = \int_{-\infty}^{\infty} f_i(x+r) f_i(x) \, dx$$

where  $f_i(x) = 1$  if phase *i* is located at *x*, and  $f_i(x) = 0$  otherwise.

Equivalent to overlaying a displaced image on an original and measuring the overlapping areas of the same phase for different displacements.

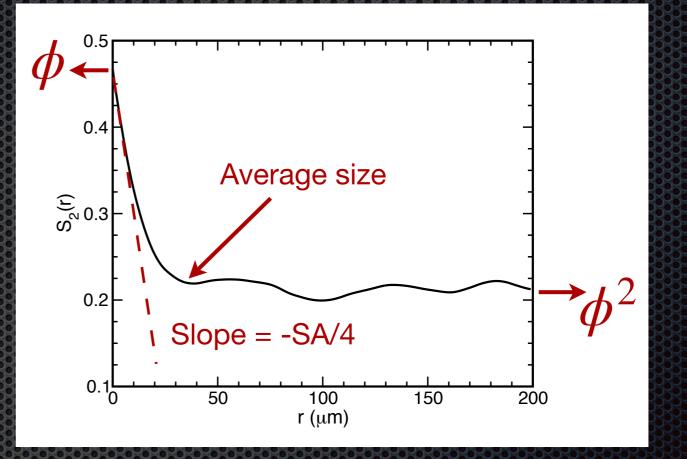


 $S_2(r) =$  Probability that second dart lands in same phase as the first dart

Features of S<sub>2</sub>:

- $S_2(0) = vol frac$
- $\Im$  S<sub>2</sub>( $\infty$ ) = (vol frac)<sup>2</sup>
- Slope at r = 0 is -1/4 of the specific surface area
- First minimum is average domain size
- Is the same in 2D and 3D for homogeneous, isotropic structures!

J.G. Berryman and S.C. Blair, *J. Appl. Phys.* **60** [6] 1930-1938 (1986)



We can use S<sub>2</sub> measured on 2D images to construct 3D images with the same statistical features

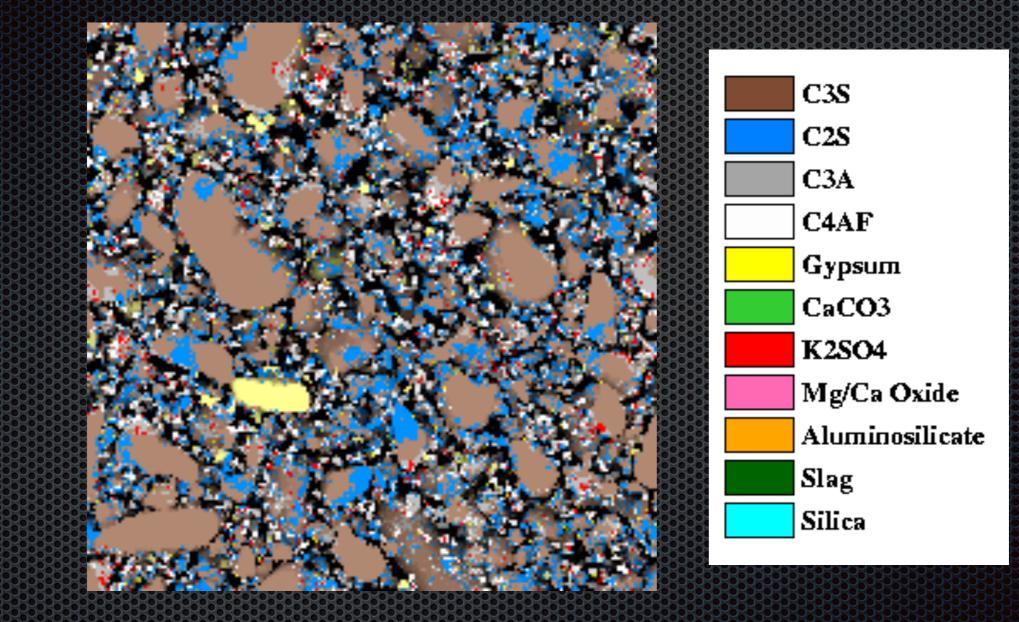


- 1. Make a 3D box of desired size populated with Gaussian noise
- 2. Filter the Gaussian noise image with S<sub>2,i</sub>(r) to create a correlated noise image corresponding to distribution of a phase i
- 3. Overlay the correlated noise image onto the generic particle image, applying the correlated noise only within the particles (this is done in the distrib3d function of the C program **genmic.c**)



- 4. Because correlation structure is dependent on which phase is considered, execute the algorithm in five passes:
  - a. separate into silicates and aluminates/alkali sulfates, then
  - b. separate aluminates and alkali sulfates
  - c. separate silicates into C<sub>3</sub>S and C<sub>2</sub>S, then
  - d. separate aluminates into C<sub>3</sub>A and C<sub>4</sub>AF
  - e. separate alkali sulfates into K<sub>2</sub>SO<sub>4</sub> and Na<sub>2</sub>SO<sub>4</sub>
- 5. After each pass, use a surface smoothing algorithm between the two modified phases until correct surface area is obtained. This is done in the function sinter3d in the C program **genmic.c**





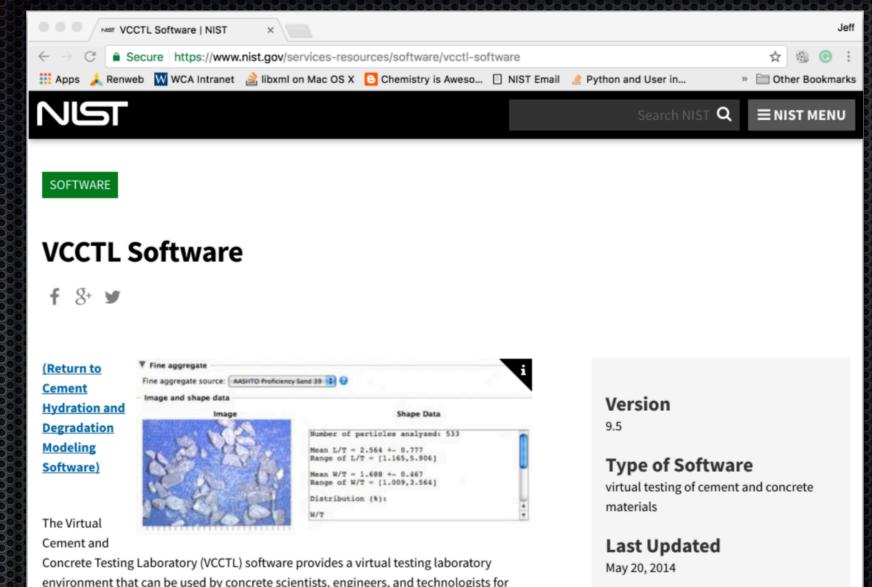


Material Inventory 😨	
Edit or create a cement	
Name: cement140 🛟 🕜 Upload data from a ZIP file for the cement:	Browse
🕨 Cement data 😨	
Mass fractions of sulfates Dihydrate 0.0039 Hemihydrate 0.022 Anhydrit	e 0.016
Cancel Save Save as Delete	
Edit or create a fly ash	

Step 1: Prepare mix (	_		Curing Conditions 🕢
Binder 🕜			
Choose a cement: cem	ient140 🛟		Conditions:
Modify phase distribution	ution in the <i>cli</i>	nker	<ul> <li>isothermal</li> <li>semi-adiabatic</li> </ul>
Modify calcium sulfat	te amounts in	the <i>cement</i>	adiabatic
,			Initial temperature: 25.0 °C
Add SCM to the bind	er		Aggregate
Mix 🕜			
	Mass fraction	Volume fraction	Aging 🕜
Binder	0.1724	0.1307	Hydrate for 28.0 days
Water	0.0776	0.1897	Or stop at degree of hydration: 1.0
Water/Binder ratio	0.45		Use time conversion
Add Coarse Aggregate	0.30	0.2658	Time conversion factor 3.5E-4 h/cycle <sup>2</sup>
Change properties			<ul> <li>Use a calorimetry file</li> <li>Use a chemical shrinkage file</li> </ul>
🗹 Add Fine Aggregate	0.45	0.4138	Saturation conditions 😨
Change properties			saturated sealed
Air		0.04	

### Cement Characterization Workshop, 2018

# NG



**NIST Authors** Jeffrey W. Bullard

environment that can be used by concrete scientists, engineers, and technologists for virtual testing of cement paste and concrete materials. With this software the user can

- · create virtual materials, using carefully characterized cement powders, supplementary cementitious materials, fillers, and aggregates;
- simulate the curing of these materials under a wide range of conditions; and

## MicroChar



## Analyzing Segmented Microstructure Images



## What you need...

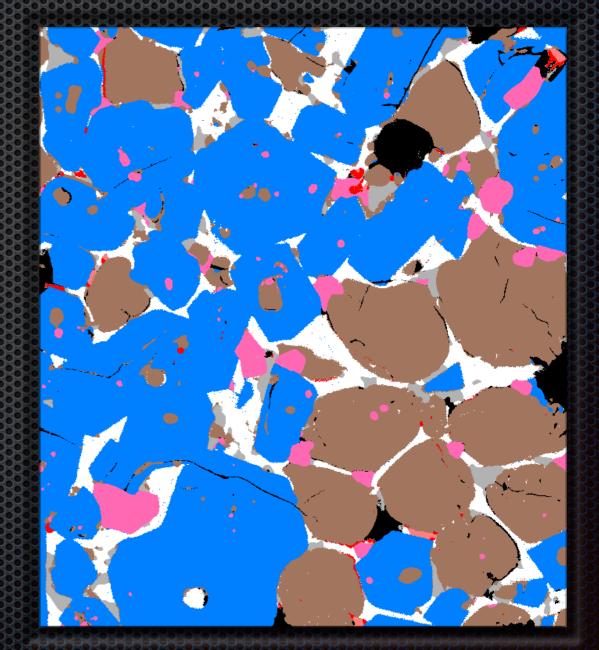
- An indexed image of a segmented micrograph (clinker or cement)
- Knowledge of the phase assigned to each index
   (1 = alite, ... 9 = void)

9	9	9	9	9	9	9	9
<b>X</b>	9	9	9	9	9	9	9
	9	9	9	9	9	9	9
ŏ	9	9	9	9	9	9	9
	9	9	9	9	9	9	9
ŏ	9	9	9	9	1	1	1
	1	1	1	1	1	1	2
ŏ	2	2	2	3	3	3	9
	9	9	9	9	9	9	9
Ö	9	9	9	9	9	9	9
	9	9	9	9	9	9	9
	9	9	9	9	9	9	9
ŏ	9	9	2	2	2	2	2
	2	2	2	9	9	9	9
ŏ	9	9	9	9	9	9	9
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ĕ	9	9	9	9	2	2	9
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	9	9	9	9	9	9	9
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	9	9	9	9	9	9	9
ŏ	9	9	9	9	9	9	9
	9	9	9	9	9	9	9
ŏ	2	2	2	2	2	2	2
	2	2	2	2	2	2	2
	2	2	2	2	2	2	9
	9999912999929999999999999999999922291	999999129999929999999999999999999922291	9999912999922999999999999999999922291	99999913999929999999999999999999922291	9999911399992999299999999999999999922211	9999911399992999299999999999999999922211	9999912999929999999999999999999999999922911
	1	1	1	1	1	1	1

## What MicroChar Does (1)



- Removes interior voids and cracks (clinker only)
- Counts pixels to get area of each phase
- Divide counts by total to get area fraction of each phase
- Area fraction = 3D volume fraction if microstructure isotropic



## What MicroChar Does (2)



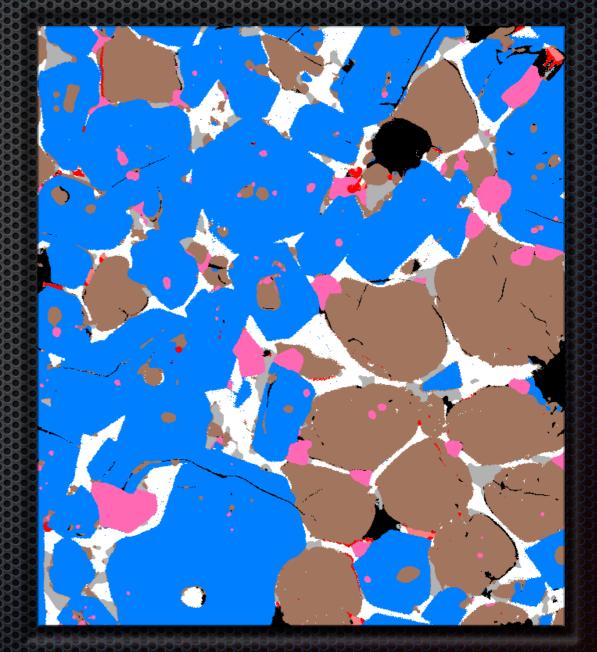
- Multiplies each phase volume fraction by the published value of its density to get scaled mass
- Divide each scaled mass by total mass to get phase mass fractions

Phase	Formula	Density (kg m <sup>-3</sup> )
Alite	C <sub>3</sub> S	3210
Belite	$C_2S$	3280
Aluminate	$\bar{C_3A}$	3038
Ferrite	$C_4$ AF	3730
Arcanite	$K_2SO_4$	2662
Thenardite	$Na_2SO_4$	2680
Gypsum	$CaSO_4 \cdot 2H_2O$	2320
Lime	CaO	3310
Calcite	CaCO <sub>3</sub>	2710
Periclase	MgO	3780
Am. Silica	SiÕ <sub>2</sub>	2650

## What MicroChar Does (3)

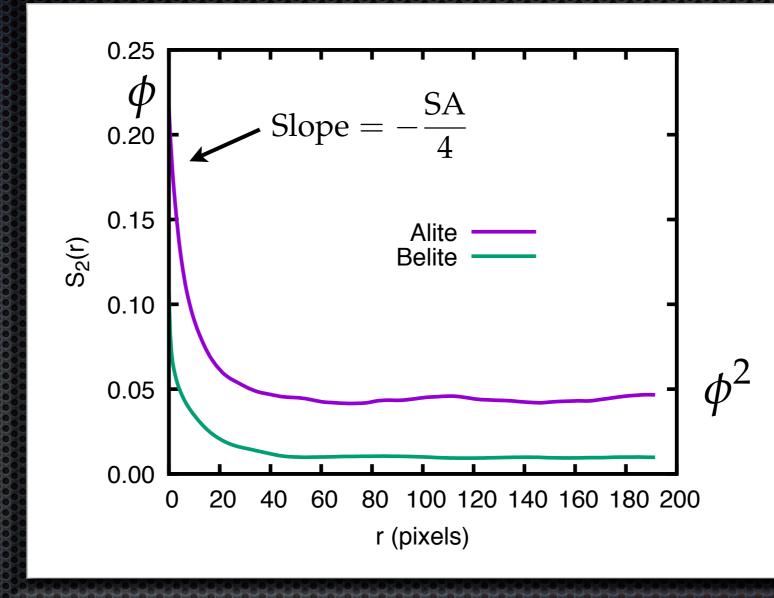


- For each phase, count number of its pixels that are next to a different phase (border), to get the phase's scaled perimeter
- Divide each scaled perimeter
   by the total scaled perimeter to
   get each phase's perimeter
   fraction
- Perimeter fraction = 3D surface area fraction if microstructure isotropic





## **Autocorrelation Functions (Optional)**



## MicroChar Download

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A	pps		LaTeX	🗎 NIST	Cement Chemistry	🗎 Christian	🗎 Linux/Unix	🗎 Databases	🗎 Machine Learning	🗎 Software Develop	Official Journal Ab	🗎 Python	»
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#### MicroChar

f G+ 🕑

#### (Return to Cement Hydration and Degradation Modeling Software)

Accurate quantitative data on cement and clinker microstructure images can prove valuable for monitoring and controlling the manufacturing of cement-based powders. Furthermore, quantitative characterization of microstructure is an essential input to microstructure-based

	Contraction of the second					
	olution: 0.5		µm/pix	œl		
Calculat	e Correlation	1 Functions				
VCCTL P	lackage	VCCTL Name:	mycement			
Gypsum:	2.0	g / 100 g				
Gypsum: Bassanite: Anhydrite:	2.0	g / 100 g g / 100 g g / 100 g	9	PSD File:	mycement.p	osd [
	2.0 0.00	g / 100 g	9			g / 100 g
Bassanite: Anhydrite:	2.0 0.00 0.1	g / 100 g	3	0.02	]	
Bassanite: Anhydrite: Na2O: K2O:	2.0 0.00 0.1	g / 100 g g / 100 g	9 9 Readily Soluble:	0.02	]	g / 100 g
Bassanite: Anhydrite: Na2O: K2O:	2.0 0.00 0.1 0.2 My VCCTL o	g / 100 g g / 100 g	9 9 Readily Soluble:	0.02	]	g / 100 g

computer models of cementitious material processing and properties. This document describes the use and operating principles of MicroChar, a computer application for automatically calculating a range of microstructural properties from an indexed 2D image. Among the properties calculated are the volume fraction, mass fraction, and surface area fraction of each phase in the image, as well as two-point correlation functions for quantifying the spatial distribution of the phases throughout the structure. The application also enables the user to package the data obtained on cement powders for

Type of Software Executable

NIST Authors Jeffrey W. Bullard

#### System/Platform Requirements

The following requirements must be met for successful installation and running of MicroChar:

- Operating system: Microsoft Windows 7\* or Mac OS X 10.9\* or later. Other versions of Windows, such as XP, Vista, or Windows 8, or earlier versions of Mac OS X may or may not be compatible with MicroChar.
- A minimum of 2 GB RAM and minimum free hard disk space of 1 GB for long-term use of thesoftware.

